NASA Invention Case No.: MSC-23277-1 *PATENT*

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant: Dennis R. Morrison
Application No.: 10/734,753 Group No.: 2877
Filed: December 9, 2003 Examiner: Nguyen, Tu T.
For: Microparticle Analysis System and Method Confirmation No.: 1973

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

AFFIDAVIT UNDER 37 C.F.R. § 1.131

Dennis R. Morrison, being duly sworn, depose and say that:

1. I am the sole inventor of claims 1, 2, 4-17, and 25-28 of the above-identified patent application.

2. I was previously employed by (and have now retired from civil service for) the National Aeronautics and Space Administration (NASA) at NASA Johnson Space Center in Houston, Texas.

3. The primary reference cited in the prosecution of the instant patent application is the United States Patent Application Publication entitled "Method for Automated Screening of Cervical/Endocervical Malignant and Premalignant Epithelial Lesions Using Flow Cytometry with HPV DNA Fluorescent In-Situ Hybridization (FISH) Technology," publication number US 2004/0260157 A1, publication date December 23, 2004, which claims the priority benefit of U.S. Provisional Patent Application Serial No. 60/480,518 filed June 20, 2003.

4. Before June 20, 2003, I completed the invention defined by claims 1, 2, 4-17, and 25-28 of the above-identified patent application in the United States of America.

5. In support of my assertion that I completed the invention defined by the instant claims in the United States of America before June 20, 2003, I submit herewith and attach hereto Exhibit A, which is a redacted photocopy of the fifteen-page Disclosure of Invention that I prepared and submitted before June 20, 2003, to the Patent Counsel of NASA Johnson Space Center located in Houston, Texas, and that was received therein before June 20, 2003. Exhibit A is a redacted photocopy because dates have been blocked off. All of the dates redacted in Exhibit A are before June 20, 2003.

6. The Disclosure of Invention contains a written description of the subject matter claimed in the present invention and establishes my conception of the invention defined by claims 1, 2, 4-17, and 25-28 of the above-identified patent application in the United States of America before June 20, 2003.

7. In further support of my assertion that I completed the invention defined by claims 1, 2, 4-17, and 25-28 in the United States of America before June 20, 2003, I submit herewith and attach hereto Exhibit B, which is a photograph of a prototype of a device or system for analyzing microparticles in laminar flow through a chamber in accordance with the invention defined by claims 1, 2, 4-17, and 25-28 of the above-identified patent application. The photograph of Exhibit B was taken before June 20, 2003.

8. The system shown in Exhibit B was built, assembled, and tested for its intended purpose at the facilities of Filter Flow Technology, Inc., located at 122 Texas Avenue in League City, Texas, before June 20, 2003. This same system shown in Exhibit B also worked for its intended purpose of analyzing microparticles in laminar flow through a chamber before June 20, 2003.

9. Exhibits A and B establish reduction to practice of the invention defined by claims 1, 2, 4-17, and 25-28 of the above-identified patent application in the United States of America before June 20, 2003.

Further deponent sayeth not.

Dennis R. Morrison

Dennis R. Morrison

STATE OF TEXAS,
COUNTY OF HARRIS, to wit:

Sworn to and subscribed before me in the aforesaid City and State by Dennis R. Morrison this 19th day of October, 2006.

Felicia Ann Saenz

Notary Public

My commission expires:

May 12, 2007

STATE OF TEXAS

FELICIA ANN SAENZ
NOTARY PUBLIC, STATE OF TEXAS
MY COMMISSION EXPIRES
MAY 12, 2007

National Aeronautics and Space Administration	Disclosure of Invention and New Technology (Including Software)	Form Approved O.M.B. No. 2700-0009	DATE
		NT CONTROL No. (Official Use Only)	

This is an important legal document. Carefully complete and forward to the Patent Representative (NASA in-house innovation) or New Technology Representative (contractor/grant innovation) at NASA. Use of this report form is optional, however, an alternative format must at a minimum contain the information required herein.

In completing each section, use whatever detail deemed appropriate for a "full and complete disclosure." Contractors/Grantees please refer to the New Technology or Patent Rights - Retention by the Contractor clauses. When necessary, attach additional documentation to provide a full, detailed description.

1. DESCRIPTIVE TITLE Microcapsule Flow Sensor

2. INNOVATOR(S) (Name(s), Title(s), Phone Number(s), Home Address(es). For non U.S. citizen, include INS Form I-551 No. and expiration date. If multiple innovators, please number.)
Dennis R. Morrison, Ph.D., Senior Biotech Project scientist, Office Tel. (281)483-7123
Address: 1802 Cove Park Drive, Kemah, Texas 77565 (281)334-5757

3. EMPLOYER(S) WHEN INNOVATION MADE (Name and Division)
NASA - JSC, SD/ Medical Sciences Division

4. ADDRESS(ES) (Place of performance)
Johnson Space Center, NASA Road One, Houston, Texas 77058

5. EMPLOYER STATUS (choose one for each innovator)	6. ORIGIN (check all that apply and supply number (s))	
GE Innovator #1 ___ Innovator #3	☒ NASA In-house Org. Code SD12	UPN(S) ___
___ Innovator #2 ___ Innovator #4	☐ NASA Grant No.	UPN(S) ___
	☐ NASA Prime Contract No. ___	UPN(S) ___
GE - Government	Task No.___ Report No.	UPN(S) ___
CU - College or University	☐ Subcontractor; Subcontract Tier ___	UPN(S) ___
NP - Non-Profit Organization	☐ Joint Effort (NASA prime contractor and NASA in-house)	UPN(S) ___
SB - Small Business Firm	☐ Multiple Contractor Contribution (collaboration of prime contractor and subcontractor)	UPN(S) ___
LE - Large Entity	☐ Other (e.g., Space Act of Cooperative Agreement) No. ___	Contractor Reportable Item No.

7. NASA Contracting Officer's Technical Representative *(COTR)*	8. CONTRACTOR/GRANTEE NEW TECHNLOGY REPRESENTATIVE *(POC)*

9. BRIEF ABSTRACT (A general description of the innovation which describes its capabilities, but does not reveal details that would enable duplication or imitation of the innovation.)

The Microparticle Flow Sensor was developed and tested as a system that can identify and count tiny particles in a flowing stream as they pass through a laminar flow chamber. The device acts as a spectrophotometer by measuring the amount of wavelength-specific, transmitted or reflected light from each particle or microcapsule. It can distinguish the target microparticles from contamination or debris based on time-of-flight,trajectory, size and spectral characteristics, when the microparticles incorporate a dye or other specific tag. Real-time sofware analysis provides identification and multiple counts in less than five seconds. The system has a dedicated CCD imaging chip, process controller, pumps and laminar flow chamber, and a wavelength-specific laser or LED light source to provide incident light. More than 1000 particles can be tracked and counted at the same time. Multi-spectral scanning enables the positive identification of several different colored or tagged particles or microcapsules at the same time. Built-in software and statistical analysis provides accurate measurements and immediate calculation of the coefficient of variations(CV). The system is compact, battery operated, and can be adapted as a portable field test unit.

Exhibit A

SECTION 1 - DESCRIPTION OF THE PROBLEM OR OBJECTIVE THAT MOTIVATED THE INNOVATION'S DEVELOPMENT (Enter as appropriate: A. - General description of problem/objective: B. - Key or unique problem characteristics; C. - Prior art, i.e., prior techniques, methods, materials, or devices; performing function of the innovation, or previous means for performing function of software; and D. - Disadvantages or limitations of prior art.)

A. General Description of Problem/Objective:
The production of uniform, multi-lamellar, liquid-filled microcapsules requires precision process control systems. An accurate, on-line, measurement system is required to identify and measure selected characteristics of each microcapsule as they are produced. Microspheres or microcapsules containing certain drugs or bioactive ingredients can be identified by spectral characteristics, however, real-time analysis must be able to distinguish between the target microparticles and sediment particles or debris which are approximately the same size. Accurate counting of dyed or tagged microparticles has numerous applications in waste water processing, bulk pumping of industrial chemicals, and identification of ownership of liquid products and fuels.

B. Key or unique problem characteristics:
It is very difficult to measure many microparticles accurately when they are suspended in flowing fluids. Inaccuracies occur when measurement systems miss individual microparticles that are moving behind or in the shadow of other microparticles that are in the foreground. It is difficult to distinquish known target microparticles from debris particules, esp. in large volume flowing liquids. Dilution of dyes or taggants can be used to measure the volume of contamination, however, this usually requires samples to be taken back to central laboratories for detailed analyses. Many of the dyes used in petrochemical products have adverse environmental problems and are very difficult to assay once the liquid has been spilled and evaporated into the air.

C. Prior art & Disadvantages:
Custodial identification of bulk products often uses special dyes and requires colorimetry type assays or full scale spectrophotometric analyses to accurately measure the concentration. Limited sample analysis can be conducted using protable colorimeters, but the data is quite limited and it is almost impossible to measure several different types of dyed or tagged microparticles at once in a mixed flow.

Various resistance or capacitance detectors can count microparticles accurately, however, they cannot determine their identity based on spectral signatures unless some version of Laser Activated Flow Cytometry or similar device is used. Such devices require sophisticated laboratories.

Image detector systems can be tuned to specific wavelengths, however, they cannot accurately measure transmitted or relfected light from hundreds of microparticles while ttracking their trajectory in the flowing stream of carrier liquid.

D. Disadvantages:
Most spectrophotometer type sensors must pass the incident light through the entire width of a flowing liquid stream in order to accurately measure the concentration of drug or dye. Snorkel type systems which measure dye concentrations must have a clear, unobstructed view of the entire light path, therefore, debris particles interfere with accurate measurements and must be eliminated from the data. It is very difficult to measure the mixed flow of two or more dyed fluids to accurately calculate the percentage composition of several different manufacturer's products.

Most imaging or optical measurement systems cannot accurately measure drug or dye concentration in individual small (10-20 micrometer) microparticles as several hundred or a thousand pass by the viewing window. They also cannot track the trajectory when the velocity is on the order of 40 - 50 micrometers per minute and they cannot distinquish target microparticles from debris.

SECTION II - TECHNICALLY COMPLETE AND EASILY UNDERSTANDABLE DESCRIPTION OF INNOVATION DEVELOPED TO SOLVE THE PROBLEM OR MEET THE OBJECTIVE *(Enter as appropriate; existing reports, if available, may form a part of the disclosure, and reference thereto can be made to complete this description: A. - Purpose and description of innovation/software; B. - Identification of component parts or steps, and explanation of mode of operation of innovation/software preferably referring to drawings, sketches, photographs, graphs, flow charts, and/or parts or ingredient lists illustrating the components; C - Functional operation; D. - Alternate embodiments of the innovation/software; E. - Supportive theory ; F. - Engineering Specifications; G. - Peripheral equipment; and H. - Maintenance, reliability, safety factors.)*

A. The present invention was designed as a microprocessor controlled Microcapsule Flow Sensor (MFS) which can identify specific microparticles or microcapsules and count them accurately as they pass abreast through a thin laminar flow chamber.

B. The MFS is composed of four major subsystems: 1) A wavelength specific light source, 2) a thin, laminar flow chamber, equipped with optical viewing ports, , 3) a CCD-type detector array and magnification lens(es), and 4) a microprocessor system with special software which can simultaneously measure and plot the location of hundreds of targets in real time.

B1. MFS Prototype: The MFS consists of a Laminar Flow Chamber constructed of polycarbonate plastic with a clear glass wall. Light was provided by a 1 mW laser tuned to 625 nm or high intensity LED lights with a peak emission of 620 nm. For the intial tests the flow chamber was approximately 50 um thick, and the flow rate through the chamber was 0.16 ml/minute. The chamber was modified to a new depth of approximately 20 um and shown to have superior resolution. A high resolution, digital camera (Kodak Model 1.4i) with a 6x magnification lens is coupled with a 400 MHz Pentium III computer and custom software to capture sequential images of microcapsules as they move horizontially through the chamber. Special software has been developed to: 1) locate individual microcapsules, 2) measure the transmitted light intensity to distinguish specific dyed micrpcapsules, 3) plot the exact location of each microcapsule on a 650 x 500 pixel array, 4) track the trajectory of individual microcapsules, and 5) calculate the sedimentation rate of each microcapsule or particle.
B2 Operations: A sample of fluid containing the target microparticles is pumped slowly (~100-200 ul/min) through the laminar flow chamber, while incident light is focused through or across the chamber. Images of the microparticles are rapidly acquired by the CCD imaging system under control of the microprocessor. The intensity of the transmitted or reflected light from each microparticle is measured, then selected values above a threshold are recorded according to the exact pixel location in the image array. Time of flight of individual microparticles is calculated, then the trajectory determined, according to a specific algorythm. Identification of the a target microparticle or microcapsule is accomplished by comparing the transmitted light received on an adjacent pixel, then subtracting the light level recorded for the target as it passes from pixel to pixel. Solid debris particles are distinguished by size, shape, and the fact that they will absorb or reflect more light than the taggant contained in the target microparticles.

SECTION III - UNIQUE OR NOVEL FEATURES OF THE INNOVATION AND THE RESULTS OR BENEFITS OF ITS APPLICATION (*Enter as appropriate: A. - Novel or unique features; B. - Advantages of innovation/software; C. - Development or new conceptual problems; D. - Test data and source of error. E. - Analysis of capabilities; and F. - For software, any re-use or re-engineering of existing code, use of shareware, or use of code owned by a non-federal entity.)*

A. Novel Features:

The MFS uses a low shear, laminar-flow chamber, which forces microcapsules to move across the chamber abreast so that each microcapsule can be detected, tracked separately, and the specific light absorption measured accurately using automated image capture and analysis. A magnification lens system is used so that each microcapsule image is equal to or slightly larger than the pixel size of the CCD imaging system. Microcapsule size & shape also can be determined to help distinguish the tagged microcapsules from particulate debris. A single CCD imaging system can be used with a moveable front surface mirror system to capture images of the same microcapsule at multiple wavelengths, thus providing multi-spectral absorption data on each individual microcapsule.

B.Advantages.

Each dyed or tagged microcapsules can be measured accurately to determine:1)specific (spectral) identity of each type of microcapsule in a mixture, 2) precise concentration in the sample fluid, 3) trajectory in a gravity field (thus the density of each target microcpasule can be calculated), 4) true microcapsules from particulate debris or bubbles within the sample stream. The MFS can be used to measure microcapsules in discrete samples. It also can be adapted (with a slip-stream type, flow through, sampling subsystem) to make real-time measurements of microcapsules flowing in a pipeline or other flowing liquid system.

Software: The custom software enables accurate determination of the exact pixel location of each microcapsule at any sample (image) time, the intensity of the transmitted light passing through each microcapsule, and a figure of merit related to the size and shape of each target particle. The software also can eliminate false positive targets, targets moving at different velocities, and monitor the trajectory to distinguish the true microcapsules from buoyant or sedimenting false targets (non-microcapsules).

C. Development or New Conceptual Problems:

A special purpose, laminar flow chamber, with an optical window had to be developed. The rapid imaging system had to be matched with the magnification system and a very slow pumping system that created a low flow rate of 150-200 microliters/min through a chamber only 20-50 microns deep. The incident light and focal plane had to be matched precisely to have each microcapsule in focus for the multiple measurements, since images were captured at a rate of more than 100 per second.

D. Test Data:

Test 1. Six (6) micron diameter microcapsules containing Cibacron Blue P2-R dye (lambda max = 575 nm) at a concentration of 9 x 10-3 Molar were found to absorb approximately 7% of the light at 620 nm. Individual microcapsules could be detected and tracked within 3-5 seconds at velocities through the chamber of approximately 100 microns/sec.

Test 2. Tests with ten (10) micron Ciba microcapsules containing Puricolor ABL-9 dye (lambda max=630 nm) showed that individual microcapsules could be identified and tracked within 1-3 seconds at velocities of up to 144 microns/sec. Sedimentation velocities could be measured at rates of 32 microns/sec for these particles when the flow in the chamber was stopped. Ref. Test Report # CSC-001, dated , submitted to Ciba Specialty Chemicals

Tests 3 and 4. The MFS laminar flow chamber was modified to reduce the thickness to approximately 20 microns. A small diaphram pump with a slip stream control allowed suspensions of microcapsule to be pumped through the chamber at 160 ul/min. Puricolor ABL-9 microcapsules (Ciba Lot 1868, mean diameter of 9.9 microns) were measured in aqueous suspension at trajectories of approximately -35 degrees from horizontal. The software also was tested to eliminate the non-moving false targets and to plot the trajectories of the test microcapsules. Ref. Test Data
and

E. Analysis of capabilities:

The initial tests have clearly demonstrated that dyed microcapsules of a size range of 6 microns or larger can be accurately identified, counted, and tracked during flow through a 3 mm x 3 mm window in a laminar flow chamber using the prototype Microcapsule Flow Sensor. At a total magnification of 45x these microcapsules can easily be visualized on a computer monitor to confirm the digital imaging data.

F. RE-engineering of existing code: The special software developed by Non Linear Optics for this prototype MFS needs to be modified to include an automated macro that will eliminate artifacts, non-moving targets, and plot the trajectory and calculate the density of candidate targets.

SECTION IV - SPECULATION REGARDING POTENTIAL COMMERCIAL APPLICATIONS AND POINTS OF CONTACT (*including names of companies producing or using similar products)*

No known industrial company are producing this type of technology.

Current contacts with potential licensees or users:

John Carter, PetrAmec, Inc., 411 Trails Court, Houston, TX 77024, (713)973-2114 (Letter of intent)
Geoff Payne, Ciba Specialty Chemicals.USA, Inc., Water Treatments, 2301 Wilroy Road, Suffolk, Va., (757)538-3700
Filter Flow Technology, Inc., 122 Texas Avenue, Leaque City, TX 77573 (281)332-3438
Process Control Outlet, Inc., 5517 East Road, Baytown, TX 77571 (281)421-1321

10. ADDITIONAL DOCUMENTATION (Include copies or list below any pertinent documentation which aids in the understanding or application of the innovation (e.g., articles, contractor reports, engineering specs, assembly/manufacturing drawings, parts or ingredients list, operating manuals, test data, assembly/manufacturing procedures, etc.))

TITLE	PAGE	DATE
Microcap Flow Sensor-Dwg	1	
Multiple-Color Flow Sensor	1	
Report CSC-001 - MeasureCiba Dye Microcapsules	7	-----

11. DEGREE OF TECHNOLOGICAL SIGNIFICANCE (*Which best expresses the degree of technological significance of this innovation?*)

☐ Modification to Existing Technology ☒ Substantial Advancement in the Art ☐ Major Breakthrough

12. STATE OF DEVELOPMENT

☐ Concept Only ☐ Design ☒ Prototype ☐ Modification ☐ Production Model ☐ Used in Current Work

13. PATENT STATUS (Prior patent on/or related to this innovation)

☐ Application Filed Application No. __ Application Date
☐ Patent Issued Patent No. Issue Date

14. INDICATE THE DATES OR THE APPROXIMATE TIME PERIOD DURING WHICH THIS INNOVATION WAS DEVELOPED (i.e. conceived, constructed, tested, etc.)

Conceived: Laminar Flow Chamber tested Microcapsule Flow Sensor Drawing -
Conceptual MFS design - as part of the Pulsed-Flow Multi-lamellar Encapsulation System (Dwg.)
Complete MFS system Constructed & Tested: October - - using Microcapsules from Ciba Specialty Chemicals and PetrAmec,Inc.

15. PREVIOUS OR CONTEMPLATED PUBLICATION OR PUBLIC DISCLOSURE INCLUDING DATES (Provide as applicable: A. - Type of publication or disclosure, e.g., report, conference or seminar, oral presentation; B.- Disclosure by NASA or Contractor/Grantee; and C. - Title, volume no., page no., and date of publication.)

Demonstration test report to Ciba Specialty Chemicals, Inc. and PetrAmec, Inc. Houston, Texas,
No public disclosures

16. QUESTIONS FOR SOFTWARE ONLY

(a.) Using outsiders to beta-test code? ☐ YES ☒ NO If yes, done under beta-test agreement ☐ YES ☐ NO
(b.) Modifications to this software continue by civil servant and/or contractual agreement? ☒ YES ☐ NO
(c.) Previously copyrighted? ☐ YES ☐ NO ☒ UNKNOWN If copyrighted, then by whom? Boulder Non-Linear System
(d.) Were prior versions distributed? ☐ YES ☒ NO If Yes, supply NASA or Contractor contact: __
(e.) Contains or is based on code owned by non-federal entity? ☒ YES ☐ NO ☐ UNKNOWN
If yes, has a license for use been obtained? ☐ YES ☒ NO ☐ UNKNOWN
(f.) Has the latest version been distributed without restrictions as to use or disclosure for more than one year?
☐ YES ☒ NO ☐ UNKNOWN If yes, date of disclosure: __

17. DEVELOPMENT HISTORY

STAGE OF DEVELOPMENT	DATE (M/Y)	LOCATION	IDENTIFY PERSONS OR RECORDS SUPPORTING FACT 17A-17E
a. First disclosure to others		NASA-JSC	John Carter, PetrAmec, In
b. First sketch, drawing, logic chart or code		NASA-JSC	Clarence Penner, Wyle
c. First written description		Houston, Tx	PetrAmec & Ciba Specialty
d. Completion of first model of full size device (invention) or beta version (software)		FFT-122 Texas Av,League C	Tod Johnson, Allen Moore
e. First successful operational test (invention) or alpha version (software)		FFT-122 Texas Av,League C	Tod Johnson, Allen Moore

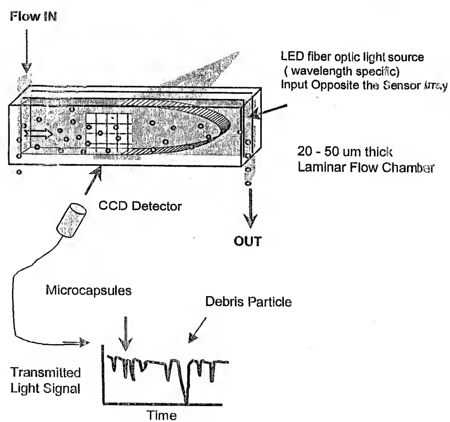
f. Contribution of innovators (*if jointly developed, provide the contribution of each innovator*)
Dennis R. Morrison -100%

g. Indicate any past, present, or contemplated government use of the innovation
Contemplated use - monitoring microcapsules made by various manufacturing techniques; detector in a feed-back control system

18 . SIGNATURE(S) OF INNOVATOR(S) DATE(S)

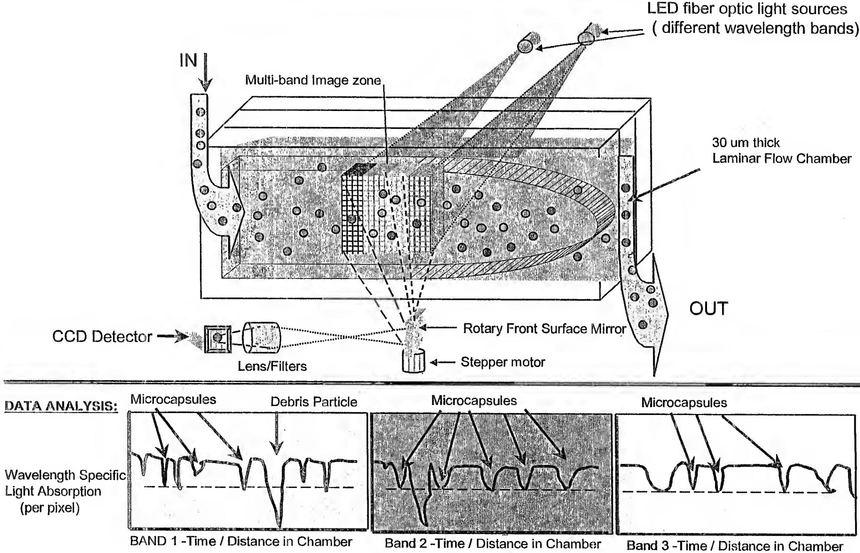
TYPED NAME AND SIGNATURE (*INNOVATOR #1*)	DATE	TYPED NAME AND SIGNATURE (*INNOVATOR #2*)	DATE
Dennis R. Morrison			
TYPED NAME AND SIGNATURE (*INNOVATOR #3*)	DATE	TYPED NAME AND SIGNATURE (*INNOVATOR #4*)	DATE
TYPED NAME AND SIGNATURE (*Witness #1*) Heather A. Lawson	DATE	TYPED NAME AND SIGNATURE (*Witness #2*)	DATE
APPROVAL NASA Tech / TYPED NAME	SIGNATURE		DATE

MICROCAPSULE FLOW SENSOR
Laminar Flow Chamber



SD12/ D.R. Morrison

MULTIPLE COLOR MICROCAPSULE FLOW SENSOR



REPORT # CSC-001

MEASUREMENT OF CIBA DYE MICROCAPSULES

Submitted by

PetrAmec, Inc.

And

NASA – Johnson Space Center,
Process Control Outlet, Inc.
Filter Flow Technology, Inc.

Houston, Texas USA

TESTING OF CIBA DYE MICROCAPSULES

Background:

Microencapsulation of different density fluids has been a part of the advanced technology development projects at the NASA –Johnson Space Center since . In addition to development of Multi-lamellar microcapsules, External-Triggered microcapsules, and space flight experiment hardware systems, the recent efforts have centered around the development of a microprocessor controlled Microcapsule Flow Sensor (MFS) which can identify specific microcapsules and count them accurately as they pass abreast through a thin laminar flow chamber.

PetrAmec, Inc. has identified several commercial applications for measurement of neutrally-buoyant , microcapsules, containing specific dyes or other taggants, to determine ownership/custody, and concentration in storage tanks or flowing pipelines. PetrAmec has concluded that the NASA-MFS prototype is the system which can be modified so that it can identify Ciba microcapsules containing selected dyes with a unique spectral signatures and distinquish the microcapsules from particulate contamination in petrochemical. Once the feasibility has been demonstrated it is the intention of PetrAmec and Ciba Specialities, Inc. to begin a collaborative project to develop two types of units that can be marketed as part of a new inventory management system.

The NASA Principal Investigator has assembled a team of scientists and process engineers from Filter Flow Technology, Inc. and Process Control Outlet, Inc. to develop: 1) a portable, self-contained Microcapsule Flow Sensor System that can be marketed as a field test unit and 2) an On-line, By-pass type of microcapsule monitoring system which can be mounted to large pipelines.

Ciba Specialties, Inc. has developed several new solid-matrix microcapsules containing dyes which are currently marketed for mixing with petroleum fuels. This report summarizes the results of the initial testing the the Ciba dye-microcapsules using the NASA MFS system.

Materials & Methods:

Microcapsule Flow Sensor - The MFS consists of a Laminar Flow Chamber constructed of polycarbonate plastic with a clear glass wall. The flow chamber was approximately 50 um thick, and the flow rate through the chamber was 0.16 ml/minute. A high resolution, digital camera (Kodak Model 1.4I), with a 3.5x magnification lens is coupled with a 400 MHz Pentium III computer and custom software to capture sequential images of microcapsules as they move horizontially through the chamber. Special software has been developed to: 1) locate individual microcapsules, measure the transmitted light intensity, and then plot the exact location on a 650 x 500 pixel array. Light was provided by a 1 mW laser tuned to 625 nm or high intensity LED lights with a peak emission about 620 nm.

Microcapsules – Two types of Ciba microcapsules were tested in the MFS.

The first set were manufactured as lots NVR 1849 –1853 containing Cibacron Blue P2-R dye at a concentration of 9 x 10^{-3} Molar. The mean size of microcapsules in lot NVR-1853 was about 6 um in diameter using a standard Coulter counter. Using a molar extinction coefficient of 5108 L $mole^{-1}$ cm^{-1} , a 6 um microcapsule should absorb about 7% of the incident light at wavelength of 600 nm, while a 10 um microcapsule should absorb about 10% of the incident light. These microcapsules were resuspended in 87 Octane gasoline for testing.

The second set of microcapsules were manufactured as Lot #1868. These microcapsules were washed to remove the light mineral oil, then resuspended in acidic (pH 6) distilled water. Our Coulter Counter measurements showed that the mean diameter was 9.9 um. They contained Puricolor Blue (ABL-9) dye at a concentration of 25 mM., with an extinction coefficient of 1.2 x 106 L $mole^{-1}$ cm^{-1} at 630 nm.

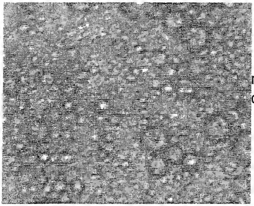
Results:

Static Tests:

1. NVR-1853 -The Microcapsule Flow Sensor was able to measure Ciba microcapsules containing Cibacron Blue P2-R dye using incident light at 630 nm. Individual microcapsules could be tracked pixel by pixel as they sedimented or as they flowed tangentially through the chamber.

2. NVR-1868 - The MFS could also measure the microcapsules containing Puricolor Blue ABL-9 dye, however, individual microcapsules were easier to visualize in the monitor using this dye. The microcapsules received in mineral oil were resuspended by gentle inversion. 5ml of the suspension was removed and placed in a 50cc conical tube. 5 ml Tween 20 was added to the microcapsule suspension and inverted to mix. This mixture was allowed to sit for 5 minutes. The microcapsules were pelleted by centrifugation at ~400 x g for 10 minutes. The supernatant was removed and the pellet of microcapsules was washed twice in 40 ml acidified water. Following the 2^{nd} wash, the microcapsules were resuspended in 50 ml acidified water and counted using a Coulter Counter.

It was determined that the suspension was at a concentration of 6.98 x 10^6 microcapsules/ml. 9.44% (6.59 x 10^5 microcapsules/ml) of these microcapsules were in the 14um range. The mean size of this suspension was found to be 9.4 um with a range of 2.8 to 37.2 um.



2

For the experiments on , the microcapsule suspension was diluted 1:10 to a concentration of ~70,000/ml.

It was found that these microcapsules sedimented rapidly in aqueous solution. This was surprising since the Malvern Mastersizer data (from Ciba) indicates that the density of the capsules is 1.000 g/ cm^3. Several microcapsules were tracked and found to be sedimenting at 32.05 um/sec.

<u>Horizonital Flowing Tests:</u>

<u>NVR-1868-</u> Microcapsules were measured and tracked with the flow chamber mounted on edge so that the flow through the chamber was horizonital. Various single microcapsules were tracked during intervals of 30 seconds. Typical microcapsules could easliy be counted at velocities of up to 144 um/sec. Different sized microcapsules moved across the image matrix at velocities that were slightly greater in the horizontal direction of flow than movement down the rows due to sedimentation.

Specific microcapsules could be identified within 1-3 seconds and the rate of movement through the chamber could be calculated within a total travel distance of less than 15 columns (~70 um) at a velocity of 100 um/sec.

Conclusions-

The MFS system can be used to identify and track either of two types of Ciba microcapsules containing Puricolor ABL-9 or Cibacron Blue P2-R dyes. The preferred size range is 10 um or greater, when using a CCD imaging system with a pixel size of 6.8 um.

Preliminary data show that the MFS can be used to accurately measure the light absorption of individual microcapsules and count them within a few seconds after they enter the image field. Light absorption by the dye corresponds to a decrease of approximately -10% of Transmitted light, that is adequate for good measurements in clear fluids.

The microcapsules containing Puricolor Blue-ABL-9 were superior to those containing Cibacron P2-R dye. Ostensibly, this was due to the increased diameter of the capsules (the increased path length of dye available to absorb the 630 nm. Light), the improved match of the dye to the wavelength of the incident light and sensitivity of the CCD chip in the camera, and finally to the increased concentration (2.8x greater).

Recommendations

1. The preliminary tests using the prototype MFS have demonstrated that the system can identify microcapsules according to dye content and size. The next technical step is to modify the software to track and determine trajectory of individual targets, so that buoyant or heavier particulates (of the same size range) can be eliminated from the measurement of the dye-tagged Ciba microcapsules.

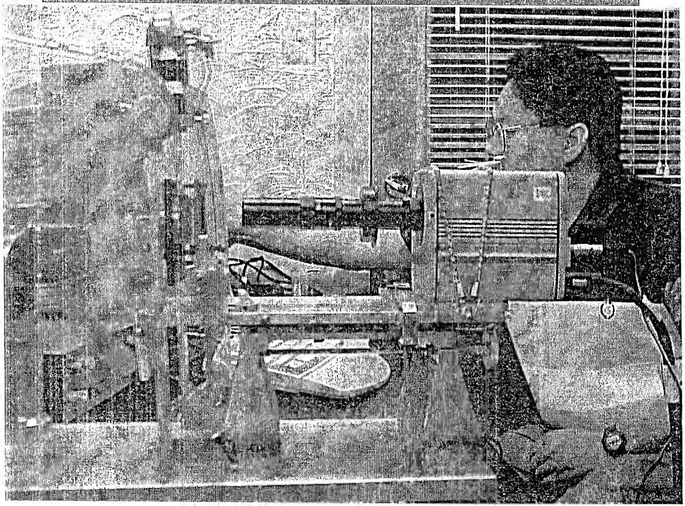
2. The density of the microcapsules is not as great an issue for measurement with the MFS system as it is for the even distribution in the fluid to be tagged so that counts per unit flow volume can be used directly to measure concentration.

3. The next series of tests will be conducted with mixtures of NVR-1868 & NVR-1873 (do not contain dye).

4. Development of the portable MFS Field Test Unit can now proceed.

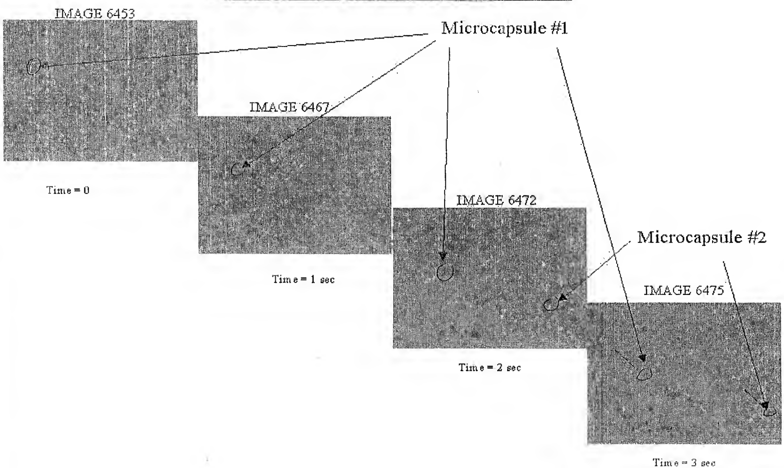
Dennis R Morrison

NASA MICROCAPSULE FLOW SENSOR PROTOTYPE

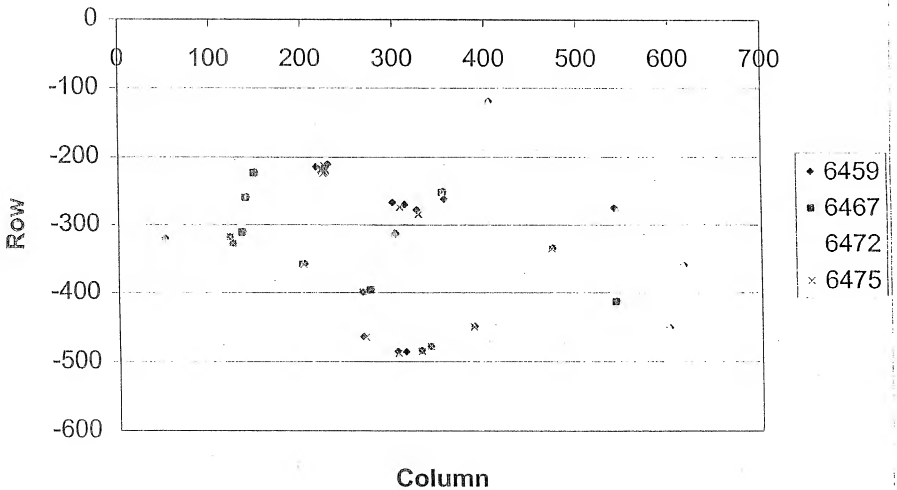


MICROCAPSULE FLOW SENSOR TRAJECTORY

D.R. Morrison



Frames 6459, 6467, 6472,6475



PetrAmec Inc. CIBA DYE-MICROCAPSULES:

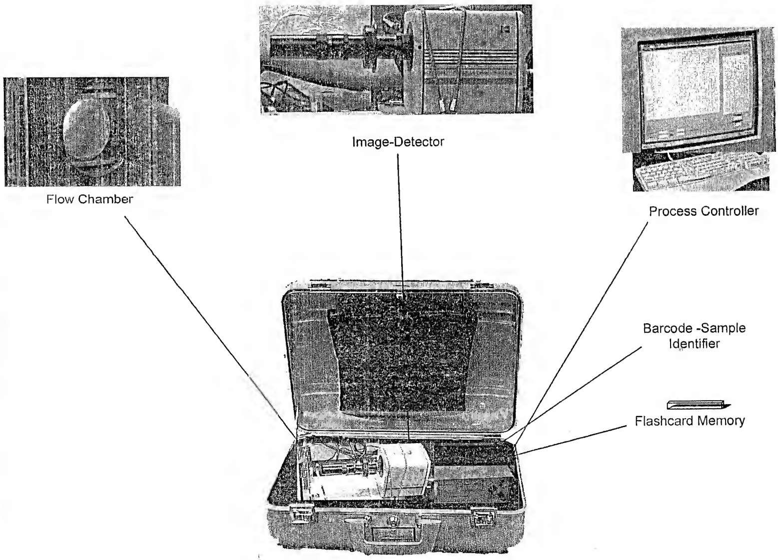


EXHIBIT B: NASA Case No. MSC-23277-1